Bonding Wires to Quantized Hall Resistors

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***Abstract* –Three different techniques for attaching wires to quantized Hall resistors with gold-germanium-nickel (AuGe/Ni) alloyed contacts were evaluated. The best quality and most robust samples were made by evaporating bonding pads that overlapped the alloyed contacts and the substrate, so that bonds could be made over the substrate rather than over the heterostructure.**

I. INTRODUCTION

OF the different techniques for making electrical contact to quantized Hall resistors made from GaAs/AlGaAs heterostructures, alloyed AuGe/Ni contacts have found wide application because they have low contact resistances, high reliability, and are easy to mass-produce. The problem of mounting these devices so that measurements can be performed on them presents many challenges. AuGe/Ni contacts are very thin and the two-dimensional electron gas (2 DEG) responsible for the quantum Hall effect is very close to the surface of the heterostructure, which makes attaching wires to these contacts without damaging them difficult. This problem is made more difficult by the extremes of temperature and fairly high stresses that the samples experience, particularly when inserted into a cold dewar. These conditions require that the wires be attached to the pads firmly enough that gusts of helium gas evolved during cooling do not cause the wires to become detached from the sample. In addition, the adhesive used to attach the sample to the header must remain adherent between room temperature and liquid helium temperature. Since quantized Hall resistance devices will be used as resistance standards over periods of many years or even decades, the adhesives used to mount the samples and the bonds between the wires and the contacts must not degrade with time. This paper describes the results of an evaluation of several different techniques for attaching samples to headers, and for attaching wires to the samples. A technique that yields clearly superior results is described.

II. EXPERIMENTAL TECHNIQUES

A number of different techniques for mounting and attaching wires to GaAs/AlGaAs quantum Hall devices with alloyed AuGe/Ni contacts have been devised and evaluated in this work. The devices were produced commercially by the Limeil GaAs Foundry of the Laboratoires d' Electronique Philips (LEP) under contract to the EUROMET Consortium [1]. These samples have alloyed AuGe/Ni contacts with a Ti/Pt/Au thickening layer over the alloyed contacts. The samples used in this study had an approximately 165 nm thick protective Si_3N_4 coating deposited over the sample and around the bonding pads. This coating did not cover the contact pads.

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Three different adhesives for attaching samples to the header were evaluated, including silicone vacuum grease, plastic film, and conductive epoxy. Wires were attached to the sample by soldering, by bonding gold wires directly to the existing gold contact pads, and by depositing enlarged gold bonding pads that permitted wires to be bonded over the substrate, rather than the heterostructure. Extensive tests on samples mounted using each of these techniques show that while all can be used to attach wires to standards-quality samples, the first two have disadvantages that make them less desirable for mounting devices that are to be used as resistance standards for long periods of time. Devices made using the third technique have proven to be the most reliable and of the highest quality. The advantages and disadvantages of each of the mounting and bonding techniques are discussed in the next section.

III. RESULTS

A. *Mounting Techniques:*

The adhesive used to attach the sample to the header must meet many demanding criteria: it must hold the sample firmly at the high temperatures at which the wires are attached (usually 150 °C to 200 °C), yet it must also remain adhesive at cryogenic temperatures, and must not be so rigid that thermally induced mechanical stresses crack the sample. Of the three adhesives used in this study, the plastic film proved unsatisfactory, for while it was easy to apply, and held the sample excellently at room temperature, it did not adhere at cryogenic temperatures. The silicone vacuum grease remained adherent at cryogenic temperatures, but was difficult to apply to very small samples and did not hold the samples firmly at high temperatures. Conductive epoxy held the samples firmly at high temperatures, and remained adhesive at cryogenic temperatures, but during curing, emitted organic vapors that condensed on the surface of the chip, and interfered with the bonding process. [2]

The best technique for mounting samples was found to be to attach the sample to a glass plate with gold pads on it using epoxy, cure the epoxy, then clean the plate with the attached sample thoroughly with solvents, attach wires to the chip, and finally mount the glass plate with the sample in the header using silicone vacuum grease. This arrangement had the added advantage that samples could be removed from the header easily without damaging the sensitive wire-bonded connections on the sample: only the wire bonds from the header pins to the glass slide, which were easily replaced, needed to be disconnected.

B) *Bonding Techniques:*

Quantized Hall resistance devices are extremely sensitive to electrically active defects in the contacts. As a consequence, great care must be taken when attaching wires to the contacts to prevent damage to them. The samples, however, experience quite severe mechanical stresses when cooled down, so the wires must be attached to the pads in a fairly robust manner.

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Three different techniques have been used to attach gold wires to LEP samples, including soldering, bonding wires directly to the AuGe/Ni pads, and bonding wires to enlarged bonding pads evaporated over the AuGe/Ni pads.

Prior to bonding, the resistivity of the sample was determined by passing current through the contacts at the ends of the devices (the "Source" and "Drain" contacts), and measuring the potentials between pairs of potential pads using a probe station. The probes were 50-μm to 75-μm diameter tungsten wires etched to sharp points. They were about 5 mm in length, and were bonded to 250-μm thick beryllium-copper strips. The force exerted by the probes on contact pads was so small that the probes did not leave any marks on the pads. Three-terminal resistance measurements were made using the circuit of Fig. 1. While in principle the contact resistances can be determined from these measurements and the spreading resistances calculated from the sample geometry and the resistivity of the sample measured previously, it was found to be difficult to do this in practice. Such measurements can, however, be used to estimate *changes* in contact resistances, for the spreading resistances should not change with processing, so that differences between the 3-terminal measurements before and after processing can be attributed to changes in the contact resistances. These resistance measurements were conducted in normal laboratory lighting conditions. Since the conductivity of the sample is a strong function of the intensity of light shining on it, slight variations in the intensity of light incident on the sample during measurements gave rise to a random variation of about $\pm 5\ \Omega$ in these measurements.

The three-terminal resistances were measured as a function of current between -100 μA and +100 μA for each of the 8 contacts (the current source and drain contacts, and each of the 6 potential pads) at room temperature before processing, after the epoxy holding the sample to the glass slide was cured, and again after wires were attached to the sample. The sample was then cooled to 1.4 K, and the contact resistances were measured under quantum Hall effect (QHE) conditions, again using the circuit of Fig. 1. [3] When the magnetic field is set to the center of the $i = 4$ plateau in the Hall voltage, the spreading resistance (measured between pairs of potential contacts on the same side of the Hall bar) is very small ($\rho_{xx} \approx 7$ mΩ at 1.4 K, in general agreement with ref. [1]), so the measured voltage indicated in Fig. 1 is essentially equal to the sum of the contact resistance and the resistances of the wires in the cryostat probe, which were about 1.4 Ω.

1. Soldering: Because of the great sensitivity of the contacts to mechanical damage, and because soldering does not mechanically disturb the contact, it would seem that this would be the most desirable technique for attaching wires to the sample. Pure indium solder was used in all experiments because of its low melting point and high ductility. Trial was made of several different "soldering" techniques, including inserting clean 25-μm diameter gold wires into molten indium beads on the pads, and coating the ends of the gold wires with a small amount of indium, and then lightly pressing the wires onto the gold pads while the sample was maintained at a temperature of about 120 °C.

Three of the EUROMET samples (two with protective Si_3N_4 coating, and one without) were mounted using these techniques. Three-terminal resistances measured at room temperature were the same after the wires were attached to the

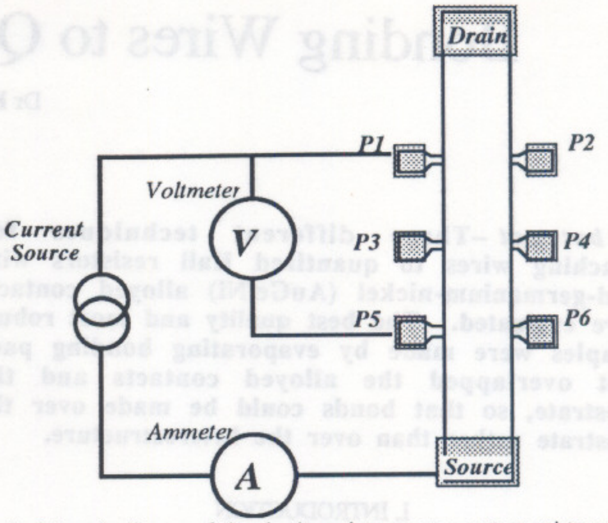

Fig. 1: Schematic diagram of the circuit used to measure contact resistances. Current enters the sample through the contact being tested (in this case the contact P1), and exits through a second contact (the "Source"), while the potential is measured between the contact being tested and a third contact (P5). At room temperature, the measured resistance, V_{15}/I, is ideally equal to the sum of the contact resistance of P1, the spreading resistance of the neck of probe P1, the spreading resistance of the channel between P1 and P5, and the wires connecting the meters and current source to the sample.

contacts as they were before, to within the uncertainty of about $\pm 5\ \Omega$, indicating negligible change in contact resistance. The contact resistances were measured under QHE conditions (1.4 K, $i = 4$ plateau) as described above. The Source and Drain (i.e., current) contacts on all samples exhibited zero contact resistance (excluding wire resistance) to within the limits of uncertainty[1] of $\pm 0.3\ \Omega$ up to currents of about 300 μA. Above this current, the samples exhibited breakdown, and the resistance of the sample (ρ_{xx}) increased rapidly, in agreement with ref. [1]. The potential contacts on all samples exhibited zero contact resistances up to currents of about 35 μA to 40 μA, above which the contact resistances began to increase rapidly, possibly due to breakdown caused by high current density in the narrow necks of the probe pads.[2]

Soldering techniques are so successful primarily because the gold-germanium contacts are never subjected to any mechanical stresses, so the contacts are not mechanically damaged during bonding. Indium readily forms intermetallic compounds with gold, so that mechanically strong bonds with low electrical resistance are formed even if the contacting surfaces are not extremely clean. The formation of these intermetallic compounds, however, also has negative consequences. We have found that gold wires attached to alloyed indium contacts on quantum Hall devices have become brittle and broken after having been stored at room temperature for periods of 10 years or more. This confirms the findings of Braun et al. [4] that even at room temperature, gold in contact with indium-containing solders forms brittle intermetallic compounds, primarily $AuIn_2$. The rate of formation of these compounds is such that the entire thickness of a 25-μm diameter wire will be

[1] The uncertainty due to random effects in the measurement was less than 0.1 Ω. The resistance of the wires connecting the sample to the measurement circuit is subtracted from the measured resistance (see Fig. 1), and the resistance of the wires in the probe is a function of the length of wire immersed in liquid helium. This varies from measurement to measurement, giving rise to an uncertainty in the wire resistance of about 0.3 Ω.

[2] Note that the critical current density in the 400 μm wide channel is about 300 μA/400 μm = 0.75 A/m; a current of 37 μA flowing through the 50 μm wide potential pad necks would produce this same current density, so currents between 35 and 40 μA would be expected to cause breakdown in the pad necks.

consumed in about 10 to 20 years at room temperature. The rate of reaction between the gold and the indium can be even higher if the gold is in the form of a thin film, as reported by Simić et al. [5]

It is therefore clear that with the passage of time, the gold wire, and more importantly, the gold in the alloyed contact will react with the indium to form intermetallic compounds. These compounds are brittle, and are likely to suffer mechanical failure when the sample is cooled to the cryogenic temperatures required to observe the quantum Hall effect. This will result in catastrophic failure of the device, for the ohmic contact formed at the AuGe-GaAs interface will have been eaten away by the indium, and completely new alloyed contacts would have to be made to the device. This technique is therefore ideal for use in testing samples that will not have to be used for long periods of time, but is unacceptable for preparing quantum Hall resistors that will have to be used as resistance standards for periods of 10 years or more.

2. *Direct Wire Bonding:* Since the contact between gold wires bonded directly to gold pads using thermosonic or compression bonding does not degrade with time, it would seem that this technique would be ideally suited to attaching wires to quantized Hall resistance standards. Bonding wires directly to the gold pads on the heterostructure, however, is an extremely delicate task. The 2 DEG responsible for the quantum Hall effect is only about 60 nm below the contact, so excessive stress applied to the contact during wire bonding will result in the formation of electrically active structural defects in the heterostructure directly beneath the metal contact, causing unacceptably high contact resistances.

The problem is made even more difficult by the fact that GaAs has an extremely low yield stress.[3] Furthermore, electrically active defects will form at pressures far below the yield stress: bonding pressures as low as 73.5 MPa have been found to create electrically active defects, *even if there was no applied ultrasonic power*. [7] Since the defects act as acceptors, they raise the resistance of the contacts, and can cause them to cease to carry current at low temperatures. In order to bond wires to the pads without damaging the contacts, therefore, the lightest possible bonding forces must be used. While application of ultrasonic energy to the bonding tool during bonding will enhance the plastic deformation of the metal wire and create a stronger bond for a given applied bonding force, the ultrasonic energy tends to greatly increase the formation of defects in the semiconductor beneath the bond, as discussed by Vidano, et al. in ref. 6. As a result, one must use the minimum possible ultrasonic energy when bonding to GaAs, and preferably use no ultrasonic energy at all. [8] Under these conditions, however, the wire will not stick to the sample unless both the wire and the bonding pad are of the highest cleanliness.

Gold wires with a diameter of 25 μm and a tensile strength of 5.9 cN and "4 % elongation" were bonded to several coated EUROMET samples. The sample was maintained at a temperature of 200 °C and no ultrasonic power was applied to the bonding tool during bonding. Since application of a force of 6 cN to a 25 μm diameter wire resting on a pad would result in a pressure on the GaAs beneath the wire of between 47 MPa and 94 MPa (depending on the area of contact between the wire and the pad), and since electrically active defects are formed at pressures as low as 73 MPa, the formation of some electrically active defects beneath the bond is practically unavoidable. In order to ensure that bonds could be made with the minimum of force the samples were carefully cleaned in solvents, and the gold wires were etched in a hydrochloric acid-hydrogen peroxide solution prior to bonding. Even with such treatment, it was not found to be possible to get the wires to adhere well to the sample when bonded with the minimum bonding force possible with our wire bonder (17 cN). Bonds were made with a bonding force of 21 cN to 24 cN. Such a low bonding force scarcely deformed the wire at all.

As expected, the room-temperature three-terminal resistances measured using the circuit of Fig. 1 after bonding were uniformly larger than the same resistances measured before bonding, indicating increases in the contact resistances. Some contacts were affected much more than others: on one sample, the wires were bonded with a force of only 19 cN, and the source and drain 3-terminal resistances increased from ≈ 4.7 kΩ to ≈ 22 kΩ (indicating an increase of about 17 kΩ in the contact resistances), while the potential probe resistances only increased an average of about 40 Ω, even though all probes on that sample were subjected to identical bonding conditions. The best samples made using this technique exhibited room-temperature contact resistances after bonding that were about 10 Ω to 30 Ω higher than before bonding.

The contact resistances were measured under QHE conditions as a function of current between -100 μA and +100 μA. Generally, the contact resistances (excluding the probe wire resistances) measured at 1.4 K and 5.06 T (center of the $i = 4$ plateau) were between zero and 36 Ω. While a few contacts exhibited constant contact resistances up to currents as high as ± 40 μA, the resistance of most others began to increase rapidly at currents as low as 0.5 μA. Other contacts, however, exhibited very large contact resistances (≈ 70 Ω) at *low* currents (≤ 10 μA), and *low* contact resistances at *higher* currents. The contact resistances were measured again at 0.3 K and 5.06 T. At this lower temperature, the majority of the potential contacts exhibited zero resistance between ± 40 μA or 50 μA and the contact resistances increased rapidly at higher currents. A number of contacts still exhibited inverted current dependence, with the contact resistance being very large (15 Ω to 20 Ω) at low currents and decreasing to vanishing values at higher currents. The reason for this behavior is not clear, but since it was not observed in any of the samples prepared with either of the other techniques, it was presumed that these current dependence effects were related to damage to the contact produced during bonding.

Thus, in spite of the extreme precautions taken during wire bonding, there was a measurable degradation in the contact resistances after bonding. While this degradation was very small at 0.3 K, and the sample could be used as a resistance standard at 0.3 K, the extremely light pressures used to bond the wires to the sample did not attach the wires sufficiently well to withstand more than one cool-down. This low bond strength, particularly when coupled with the degradation in contact resistances due to damage produced during bonding,

[3] The yield strength is a strong function of the surface condition: chemically etched GaAs has a yield strength of about 147 MPa, while mechanically polished GaAs will fracture at pressures as low as 29 MPa. [6] Pressure exerted by probes during testing of the device can create dislocations and other defects that can reduce the yield stress considerably, and it is for this reason that special precautions were taken to ensure that the probes used in testing the sample before bonding exerted only the slightest pressure on the sample.

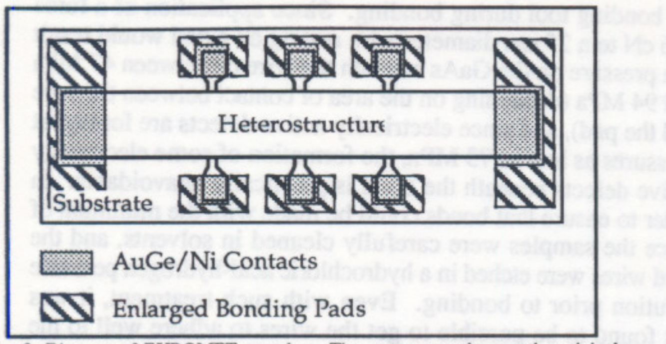

Fig. 2: Diagram of EUROMET samples. The grey rectangles are the original AuGe/Ni contacts and the cross-hatched rectangles are the gold bonding pads. The enlarged pads overlap both the AuGe/Ni contacts and the substrate, eliminating the need to perform the bonding operation over the sensitive heterostructure.

makes this technique the least desirable for mounting standards-quality quantum Hall resistors.

3. Enlarging Bonding Pads: The main problem with the previous technique was that the bonding was performed directly over the heterostructure, so that any defects generated in the gallium arsenide beneath the wire by the high forces necessary to adequately attach the wire to the gold pad, would necessarily degrade the contact. A simple solution to this problem is to enlarge the bonding pads as shown in Fig. 2 so that they extend over the semi-insulating substrate outside of the Hall bar. Wires can now be bonded over the substrate, where any damage created by the bonding will not affect the 2 DEG or the electrical quality of the contact.

Two of the LEP samples with protective silicon nitride coatings were prepared using this technique. The bonding pads were enlarged by evaporating a thin layer of chromium to promote adhesion between the gold and the substrate, followed by about 400 nm to 500 nm of gold. Wires were bonded to the enlarged pads over the substrate, using a bonding force of 32 cN (the sample temperature was 200 °C during bonding). A small amount of ultrasonic power was applied to the sample during the bonding operation, resulting in firm bonds to the pads.

The room-temperature three-terminal resistances measured after the bonding operation were in all cases identical (to within the uncertainty of ± 5 Ω) to the resistances measured before bonding. This is not surprising, for the contact pads were not subjected to any mechanical stress. The contact resistances were measured as a function of current under quantum Hall conditions (1.4 K, magnetic field at the center of the $i = 4$ plateau). In all cases, the contact resistances (excluding wire resistances) were zero to within the limits of measurement at currents less than a critical current, and increased rapidly above the critical current. The critical current for the source and drain contacts was about 300 μA, and the critical current for the potential contacts was between 30 μA and 45 μA.

The uniformly excellent quality of the contacts, coupled with the fact that the wires are very firmly attached to the sample and can therefore withstand the thermal and mechanical shocks that the samples are exposed to in normal use, make this technique ideal for use in mounting standards-quality quantum Hall devices. In addition, current experience indicates that these contacts should not deteriorate with time, permitting these samples to be used for long periods of time without requiring repairs.

IV. CONCLUSION

Soldering and wire bonding were evaluated as techniques for attaching wires to high quality quantized Hall resistors with AuGe/Ni alloyed contacts. Soldering or "indium-bonding" was the easiest to implement, but samples made using this technique potentially suffer from long-term degradation due to the formation of intermetallic compounds at the Au-In interface. The highest quality, most resilient samples were prepared by depositing gold bonding pads that overlapped the AuGe/Ni contacts and the substrate, enabling bonding to be performed over the substrate so that damage to the brittle GaAs during bonding did not affect the electrical quality of the contact. Samples prepared in this manner had the highest breakdown currents and lowest contact resistances of any of the samples made using the other techniques.

Note added in February 1995: The experiments reported in the paper were performed on EUROMET samples with a silicon nitride protective coating. Subsequent to the submission of the manuscript, the enlarged pad technique was used to mount EUROMET samples without the protective coating. The pads and bonds were found to adhere excellently, no degradation of the contacts was observed after bonding, and the samples were robust, and of the highest quality.

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